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METHOD OF OBTAINING HETEROCYCLIC POLYMERS(U) FOREIGN
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L I CHUDINA ET AL. 07 AUG 84 FTD-ID(RS)T-0795-84

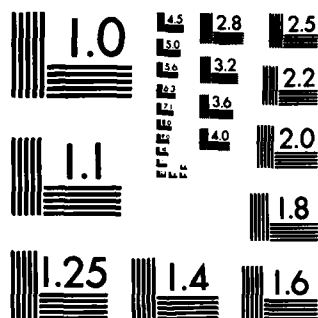
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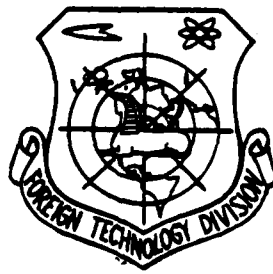
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METHOD OF OBTAINING HETEROCYCLIC POLYMERS

by

L. I. Chudina, K. N. Vlasova, et al



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EDITED TRANSLATION

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METHOD OF OBTAINING HETEROCYCLIC POLYMERS

By: L. I. Chudina, K. N. Vlasova, et al

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WP.AFB, OHIO.

U. S. BOARD ON GEOGRAPHIC NAMES transliteration SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	<i>А а</i>	A, a	Р р	<i>Р р</i>	R, r
Б б	<i>Б б</i>	B, b	С с	<i>С с</i>	S, s
В в	<i>В в</i>	V, v	Т т	<i>Т т</i>	T, t
Г г	<i>Г г</i>	G, g	У у	<i>У у</i>	U, u
Д д	<i>Д д</i>	D, d	Ф ф	<i>Ф ф</i>	F, f
Е е	<i>Е е</i>	Ye, ye; E, e*	Х х	<i>Х х</i>	Kh, kh
Ж ж	<i>Ж ж</i>	Zh, zh	Ц ц	<i>Ц ц</i>	Ts, ts
З з	<i>З з</i>	Z, z	Ч ч	<i>Ч ч</i>	Ch, ch
И и	<i>И и</i>	I, i	Ш ш	<i>Ш ш</i>	Sh, sh
Й й	<i>Й й</i>	Y, y	Щ щ	<i>Щ щ</i>	Shch, shch
К к	<i>К к</i>	K, k	Ъ ъ	<i>Ъ ъ</i>	"
Л л	<i>Л л</i>	L, l	Ы ы	<i>Ы ы</i>	Y, y
М м	<i>М м</i>	M, m	Ь ь	<i>Ь ь</i>	'
Н н	<i>Н н</i>	N, n	Э э	<i>Э э</i>	E, e
О о	<i>О о</i>	O, o	Ю ю	<i>Ю ю</i>	Yu, yu
П п	<i>П п</i>	P, p	Я я	<i>Я я</i>	Ya, ya

*ye initially, after vowels, and after ъ, ы; e elsewhere.
When written as ё in Russian, transliterate as yě or ě.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh ⁻¹
cos	cos	ch	cosh	arc ch	cosh ⁻¹
tg	tan	th	tanh	arc th	tanh ⁻¹
ctg	cot	cth	coth	arc cth	coth ⁻¹
sec	sec	sch	sech	arc sch	sech ⁻¹
cosec	csc	csch	csch	arc csch	csch ⁻¹

Russian English

rot curl
lg log

GRAPHICS DISCLAIMER

All figures, graphics, tables, equations, etc. merged into this translation were extracted from the best quality copy available.

METHOD OF OBTAINING HETEROCYCLIC POLYMERS

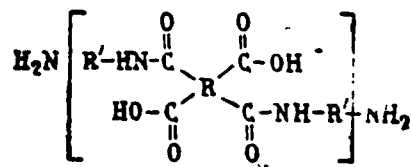
Author's certificate number 378397

Authors of invention: L. I. Chudina, K. N. Vlasova,
S. I. Litovchenko, N. P. Bordyukova and N. F. Sedova

The invention pertains to methods of obtaining thermostable, heterocyclic polymers with a cross-linked structure.

A method is known for obtaining thermostable heterocyclic polymers with a cross-linked structure on a base of dianhydrides of tetracarboxylic acids, aromatic diamines and anhydrides of unsaturated dicarboxylic acids.

The reaction of polycondensation is realized in a medium of an organic polar solvent with the interaction of the dianhydride of tetracarboxylic acid with a surplus amount of diamine, as a result of which a solution of polyamido acid with end amino groups is formed:

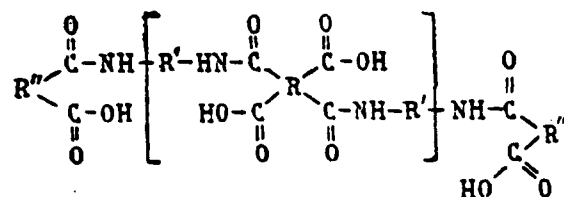


where R - radical of the tetra acid;

R' - radical of the aromatic diamine, containing one or several aromatic nuclei, connected together by heteroatoms S, O or groups of atoms SO₂, CO, CH₂.

The polyamido acids obtained are subjected to the interaction with the monoanhydride of unsaturated dicarboxylic acid at 100°C, as a result

of which polyamido acids are formed which conform to the formula:



where R'' - radical, containing no less than one double bond.

The poliamido acids obtained are converted into polyimides at 80-350°C, in this case the reactions of cyclodehydration and cross-linking take place.

However, such polymers possess a low chemical stability.

The purpose of the invention is increasing the chemical stability of the polymers.

The proposed method of obtaining heterocyclic polymers differs from the known method by the fact that as the diamines [they] use amino derivatives of phenyl-substituted oxadiazole, oxazole, benzimidazole, for example, 2,5 bis-(m-aminophenyl)-1,3,4-oxadiazole; 2,5-bis-(p-aminophenyl)-1,3,4-oxadiazole; 2,2'-bis-(m-aminophenyl)-5,5'-bibenzimidazole; 2,3-diaminobenzoxazole, and also mixtures of them with aromatic diamines.

As the dianhydrides of tetracarboxylic acids the following are used: pyromellitic dianhydride; dianhydride of :

3,3',4,4'-diphenyloxidetetracarboxylic acid;

3,3',4,4'-diphenyltetracarboxylic acid;

3,3'-4,4'-benzophenonetetracarboxylic acid;

2,3,6,7-naphthalenetetracarboxylic acid;

3,4,9,10-perylenetetracarboxylic acid; cyclopentanetetracarboxylic acid; pyrazinetetracarboxylic acid and other tetra acids.

As the anhydrides of unsaturated dicarboxylic acids they use, as an example, maleic anhydride, itaconic, cytroconic, tetrahydrophthalic.

The proposed method is realized in the following manner.

The dianhydride of tetracarboxylic acid reacts in the amide solvent (N-methyl-pyrrolidone, dimethylformamide, and others) with the diamine containing the indicated heterocycles, forming heterocyclic polyamido acid with end NH₂-groups.

The amount of dianhydride and diamine used in the course of the

reaction is selected in such a way that the excess of the number of moles of the diamine component did not exceed 0.5-1.0. Then to the solution of polyamido acid the anhydride of unsaturated dicarboxylic acid is added in a quantity which should correspond to the number of free amino groups. The resulting product is precipitated from the solution and converted to a polymer with a cross-linked structure directly in the process of formation of the item at 350-400°C and $P=270-300 \text{ kg/cm}^2$. The polymer, obtained on a base of dianhydride of 3,3'-4,4'-benzophenone-tetracarboxylic acid, 2,5-bis-(m-aminophenyl)-1,3,4-oxadiazole and maleic anhydride, has 30% lower water absorption (after holding for 24 hours in boiling water) and 25% higher alkali-resistance (after holding for 2 hours in a 10% aqueous solution of NaOH) than the known polymer on a base of dianhydride of 3,3'-4,4'-benzophenonetetracarboxylic acid, 4,4'-diaminodiphenyl ether and maleic anhydride.

Example 1. 31.0 g (0.1 m) of dianhydride of 3,3'-4,4'-diphenyl-oxidetetracarboxylic acid and 37.8 g (0.15 m) of 2,5-bis-(m-aminophenyl)-1,3,4-oxadiazole are dissolved in 220 g of dimethylformamide for 2 hours at 20-25°C. Then a solution of 2.0 g maleic anhydride in 5 ml of dimethylformamide is added to the reaction mixture, the mixture is heated to 50°C and held at 50-65°C for 2 hours. The product is precipitated in chloroform and a yellow powder with a yield of 82% is obtained. It extrudes at 300-400°C and $P=270-300 \text{ kg/cm}^2$. Strong monolithic samples are obtained in the form of pellets of a dark-brown color with $d=10 \text{ mm}$ and $h=10 \text{ mm}$.

Example 2. 31.0 g (0.1 m) of dianhydride of 3,3'-4,4'-diphenyl-oxidetetracarboxylic acid, 18.9 g (0.075 m) of 2,5-bis-(m-aminophenyl)-1,3,4-oxadiazole and 15.0 g (0.075 m) of 4,4'-diaminodiphenyl ether are dissolved for 2 hours at 20-25°C. Then a solution of 2.2 g of anhydride of cytroconic acid in 5 ml of dimethylformamide is added to the reaction mixture, the mixture is heated and held at 50-65°C for 2 hours. The product is precipitated in chloroform and a powder of a yellow color with a yield of 78% is obtained. The polymer forms by pressing at 350-400°C and $P=270-300 \text{ kg/cm}^2$, and strong monolithic samples in the form of pellets are obtained.

Example 3. 31.0 g (0.1 m) of dianhydride of 3,3'-4,4'-diphenyl-oxidetetracarboxylic acid, 16.9 g (0.075 m) of 2,3-diaminobenzoxazole and 15.0 g (0.075 m) of 4,4'-diaminodiphenyl ether are dissolved for

2 hours at 20-25°C. Then a solution of 2 g of maleic anhydride in 5 ml of dimethylformamide is added to the reaction mixture, the mixture is heated and held at 50-65°C for 2 hours. The product is precipitated in chloroform and a powder with a yield of 85% is obtained. The polymer forms by pressing at 350-400°C with $P=300 \text{ kg/cm}^2$, and strong monolithic samples of a brown color are obtained.

Example 4. 32.2 g (0.1 m) of dianhydride of 3,3'-4,4'-benzophenone-tetracarboxylic acid and 52.7 g (0.12 m) of 2,2-bis-(m-aminophenyl)-5,5'-bibenzimidazole are dissolved for 2 hours at 20-25°C. Then a solution of 2 g of maleic anhydride in 5 ml of dimethylformamide is added to the reaction mixture. The mixture is heated and held at 50-65°C for 2 hours. The product is precipitated in chloroform. A powder with a yield of 87% is obtained. The polymer is formed by pressing at 350-400°C and $P=300-350 \text{ kg/cm}^2$. Strong monolithic pellets of a dark-brown color are obtained.

Subject of invention

A method of obtaining heterocyclic polymers with a cross-linked structure by polycondensation of dianhydrides of tetracarboxylic acids with diamines and subsequent interaction of the polyamido acid obtained with the anhydride of unsaturated dicarboxylic acid, characterized by the fact that for the purpose of improving the chemical stability of the polymers obtained, as the diamines [they] use diamines which contain oxadiazole, oxazole benzimidazole cycles, and also mixtures of them with aromatic diamines.

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